

CII - Conference on Alternate Fuel & Raw Material Utilization Alternative fuel testing and analysis 24th November 2016

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Acceptance Criteria for AFR







Constituents	Limit Value
Volatile Organics Hydrocarbons	<5000 ppm
Total Organic carbon (TOC)	<1000 ppm
CaO + SiO2 + A1203 + Fe203 + SO3 (In Ash)	>80%
Chloride	<1.5%
Sulphur	<1.5%
PCB/ PCT	<5 ppm
Heavy Metals Hg Cd+Tl+Hg As+Co+Ni+Se+Te+Sb+Cr+Sn+Pb+V	<10 ppm <100 ppm <10,000 ppm

Criteria for Waste used for energy recovery



Constituents	Limit Value
Calorific Value	<2500 kcals/ kg
Ash - Liquid - Solids	<5% <20%
Chloride	<1.5%
Sulphur	<1.5%
Halogens (F+Br+I)	<1.0%
PCB/ PCT	<50 ppm
Heavy Metals Hg Cd+Tl+Hg As+Co+Ni+Se+Te+Sb+Cr+Sn+Pb+V	<10 ppm <100 ppm <2,500 ppm
рН	4 to 12
Viscocity for loquids	<100 cSt
Flash Point for liquids	<60°C



Sr. No.	Parameters to be Analyzed	Test Method/Specification
1	Voltatile Organic Compounds(VOCs)	USEPA 8260 B
2	Semi Volatile Organic Compounds	USEPA 8270 B
	(SVOCs)	
3	Total Petroleum Hydrocarbons (TPH)	USEPA 9071
4	GCV	ASTM E711
5	Proximate analysis	ASTM methods
6	Ultimate analysis	CHNS Analyser
7	METALS	USEPA 3050B and 7000 Series
8	Poly Chloro Biphenyls (PCBs)	USEPA 8082 A
9	Poly Chlorophenols (PCPs)	USEPA 8041 A
10	Organo Chlorins	USEPA 8081
11	TCLP test	ASTM D-5233 -92

Characterisation of Waste - Gas Chromatography

- Sample is introduced into system via hot, vaporising injector.
- Flow of "Carrier Gas" moves vaporised sample (i.e. gas) onto column
- Column is coated with wax type material with varying affinity for components of interest
- Components are separated in the column based on this affinity.
- Individual analytes are detected as they emerge from the end of the column through the Detector.





Characterisation of Waste - VOCs by USEPA Method 8260B



- Volatile compounds are introduced into a GC with a capillary column by a purge-andtrap method or by other methods including azeotropic distillation, vacuum distillation, head space, direct injection, etc.
- Identification of target analytes accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards.
- Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a five-point calibration curve.



Characterisation of Waste - SVOCs by USEPA Method 8270D



- The samples are cleaned & prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation and sample cleanup procedures (refer to Method 3600).
- The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.
- Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection.
- Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards.
- Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.
- The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.



Characterisation of Waste - Total Petroleum Hydrocarbons by 9071B



2.1 A representative portion of wet (as received waste is acidified with concentrated HCI and chemically dried with magnesium sulfate or sodium sulfate. Magnesium sulfate monohydrat is used to dry acidified sludges as it will combin with 75% of its own weight in water in forming MgSO C 7H O. Anhydrous sodium sulfate is used to dry soil and sediment samples. 42 2.2 After drying, the HEM is extracted with nhexane using a Soxhlet apparatus. The n-hexane extract is then distilled from the extra and the HEM is desiccated and weighed. 2.3 When necessary, a separate sample portion is evaluated for percent solids, and the dry weight fraction may be used to calculate the dry-weight HEM concentration of the soil, sediment. or waste.

Start 11.1 Determine dry weight fraction of sample. 11.2.1.1 Weigh 11.2 ls 11.2.2.1 Decant a sample of Sludge Sediment/ wet sludge sample water: mix Soil and place in sludge or sample; discard beaker. sediment/ foreign objects. soil? 11.2.2.2 Blend 11.2.1.2 with sodium Acidify to sulfate: add pH <2. to extraction thimble. 11.2.1.3 Add magnesium sulfate в monohydrate and stir. 11.2.1.5 Remove and grind solids to a fine powder.

Characterisation of Waste - PCBs by 8082 A

- Extract of the waste extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) may be subjected to a sequential sulfuric acid/potassium permanganate cleanup (Method 3665)
- After cleanup, the extract is analyzed by injecting a measured aliquot into a gas chromatograph equipped with either a narrow- or wide-bore fusedsilica capillary column and either an electron capture detector (GC/ECD) or an electrolytic conductivity detector (GC/ELCD).
- The chromatographic data may be used to determine the selected/ individual PCB congeners, or total PCBs



2-Chlorobiphenyl
2,3-Dichlorobiphenyl
2,2',3,3',4,4'4',6-Heptachlorobiphenyl
2,2',4,4',5,6'-Hexachlorobiphenyl
2,2',3',3',4,5',6,6'-Octachlorobiphenyl
2,2',3',4,6-Pentachlorobiphenyl
2,2',4,4'-Tetrachlorobiphenyl
2,4,5-Trichlorobiphenyl



PCPs by 8041 A

- 4-Chloro 3-methylphenol
- 2-Chlorophenol
- 2,4-Dichlorophenol
- Pentachlorophenol
- 2,4,6-Trichlorophenol

Organo Chlorines by 8081 Aldrin Alpha- BHC Beta-BHC Delta – BHC Dieldrin Endosulfan I (Alpha) Endosulfan II (Beta) Endosulfan Sulfate Endrin Endrin Aldehyde Endrin Ketone Gamma – BHC Heptachlor Heptachlor Epoxide Isomer B **Methoxychlor** 4,4' - DDD4,4' - DDE4 4' - DDT

Inductively coupled plasma mass spectrometry (**ICP-MS**) is a type of mass spectrometry which is capable of detecting metals and several nonmetals at concentrations as low as one part in 1015 (part per quadrillion, ppq) on non-interfered low-background isotopes.

Samples are introduced into an argon plasma as aerosol droplets. The plasma dries the aerosol, dissociates the molecules, and then removes an electron from the components, thereby forming singly-charged ions, which are directed into a mass filtering device known as the mass spectrometer.





Characterisation of Waste - Heavy Metals by USEPA 3050B/ 7000



- For the digestion of samples, a representative 1-2 gram (wet weight) or 1 gram (dry weight) sample is digested with repeated additions of nitric acid (HNO) and hydrogen peroxide and/ or hydrochloric acid (HCI)
- this digestate is filtered and the filter paper and residues are rinsed,
- The digestate is then diluted to a final volume of 100 mL.

- Metals in solution may be readily
 determined by flame (direct aspiration)
 atomic absorption spectrophotometry.
 The method is simple, rapid, and
 applicable to a large number of
 environmental samples
- With the exception of the analyses for dissolved constituents, all samples require digestion prior to analysis.
 Analysis for dissolved elements does not require digestion if the sample has been filtered and then acidified.

B U R E A U V E R I T A S

TCLP comprises four fundamental procedures:

- Sample preparation for leaching
- Sample leaching
- Preparation of leachate for analysis
- Leachate analysis

In the TCLP procedure the pH of the sample material is first established, and then leached with acetic acid. Sodium hydroxide solution at a 1:20 mix of sample to solvent. For example, a TCLP jug may contain 100g of sample and 2000 mL of solution. The leachate mixture is sealed in extraction vessel for general analytes, or possibly pressure sealed as in zero-headspace extractions (ZHE) for Volatile Organic compounds and tumbled for 18 hours to simulate an extended leaching time in the ground. It is then filtered so that only the solution (not the sample) remains and this is then analyzed.





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Thank you